LANGMUIR PROBE DIAGNOSTICS OF CONTINUOUS, ELECTRON BEAM PRODUCED PLASMA IN ARGON, NITROGEN AND THEIR MIXTURES*

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Abstract

Langmuir probe diagnostics have been carried out in the Naval Research Laboratory's Large Area Plasma Processing System (LAPPS) using a continuous, electron-beam produced plasma. The electron beam was extracted from a hollow-cathode discharge, accelerated using a high-voltage grid and a magnetic field was applied to confine the beam. The plasma was produced in a background of pure argon, nitrogen and their mixtures. The generated plasma had a high density 10^9–10^{11} cm^{-3} like the conventional discharges but a lower electron temperature (< 1 eV) which allows for the processing of energy-sensitive materials. The effect of the operating parameters including hollow cathode and extraction voltages and currents on the plasma characteristics was examined. Further tuning of the plasma properties was achieved by adjustment of the magnetic field and the gas composition.

I. INTRODUCTION

Langmuir probes are among the most commonly used electrostatic probes for plasma characterization due to their versatility, simplicity of construction, and easy data acquisition. A Langmuir probe could be just an electrode inserted into the plasma and connected in series with a variable voltage source. The plasma parameters are calculated from the current voltage (I-V) characteristics of the probe, which are obtained by varying the probe bias around the plasma potential and determining the current collected by the probe from the plasma. The plasma parameters that were of interest in this study include plasma potential, electron temperature and plasma density, which are essential for any material processing application.

The Large Area Plasma Processing Systems (LAPPS) developed at the Naval Research Laboratory, which uses magnetically collimated, high energy electron beam for plasma generation has unique characteristics that distinguish it from the conventional plasma sources used for material processing. This source offers localized plasma production, which is determined by the electron beam dimensions and allows for the independent positioning of the stage allows control of the ion flux that arrives at the material surface.

Due to the different approach to plasma production in discharges and electron beam generated plasmas, the effects of various gas-phase processes are notable. In conventional discharge sources, external electric fields are applied to heat the electrons, which in turn excite, dissociate and ionize the background gas. Due to the low species creation thresholds excitation and dissociation govern the electron loss process and only small fraction of the electron energy is spent for ion production. However when kilovolt electron beam passes through gas, ionization is the dominant process (followed by excitation and dissociation) with cross-sections similar for all gases [1, 2], which means that by adjusting the composition of the background gas control over the species production can be achieved.

Probably the most important characteristic of the electron beam generated plasmas is that the electron temperature is less than 1 eV [3]. This results in lowering of the plasma potential below the values typical for the conventional plasma sources and thus allows for bombardment of the material surface with low energy ions. This ensures low surface damaging and is particularly important for material surface activation.

Pulsed e-beam generated plasma was successfully applied in a number of material processing applications including nitriding, thin film deposition, polymer surface modification [4]. The focus of this paper however is the experimental characterization of continuous (DC) electron beam plasmas generated in argon, nitrogen and their mixtures. The electron temperature and plasma density and their spatial profiles were measured in argon. The effects of process conditions including gas pressure and magnetic field on the plasma characteristics in argon were investigated as well. The influence of gas composition was determined by comparing results in pure argon with results in pure nitrogen and argon-nitrogen mixtures. The effect of hollow-cathode voltage variation was measured

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in argon-nitrogen mixtures. It will be shown that the electron temperature in DC electron beam plasma is lower than 1 eV and the plasma density is quite high for the large span of the investigated operating conditions.

II. EXPERIMENTAL SETUP

A. Chamber design

The experimental apparatus is shown in **Figure 1**. The stainless steel chamber is 50 cm in diameter and 65 cm tall. The system vacuum was maintained by a 100 l/s diffusion pump that provides base pressures in the 5 x 10^{-6} range. Mass flow controllers were used to supply gas to the chamber. The purity of gases used in these experiments was 99.999% for argon and > 99.8 % for nitrogen. A Convectron® vacuum gauge was used to determine operating pressures. The electron beam was extracted from a hollow cathode positioned on top of the chamber and was accelerated through a slotted anode at ground potential. The beam is terminated by a final anode located approximately 50 cm away at the bottom of the chamber. The magnetic field directed along the electron beam axis was generated with a pair of Helmholtz coils. The magnetic field strength in all the experiments (except when stated otherwise) was 100 G.

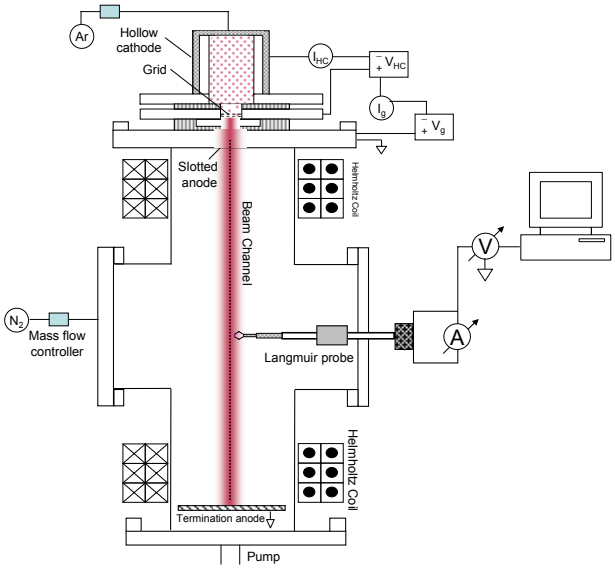

Figure 1. Schematics of the experimental apparatus

B. Hollow cathode design

The DC discharge in the hollow cathode produces electrons that are extracted through the grid and then accelerated to produce an energetic electron beam. The discharge thus determines the beam current, while the accelerating voltage determines the beam energy. The hollow cathode operates at a few hundred volts with respect to a wire-grid anode, which is held at a large negative voltage (up to – 5 kV) with respect to a grounded plate containing a rectangular slot. The hollow cathode has a rectangular cross section with 6.5 cm width, 18.5 cm length and 7 cm depth. The hollow cathode has a rectangular opening 2 cm wide and 14 cm long at the end adjacent to the wire grid anode. Electrons produced in the hollow cathode pass through a grid 1 cm wide and 10 cm long and then through a slot of the same size in a planar anode. The slotted anode is positioned approximately 1 cm from the grid and has an opening 2 cm wide and 14 cm long. The grid consisted of stainless steel mesh with 50 % transmission and openings 60 x 60 μm. The hollow cathode, the grid and the slotted anodes were made from stainless steel. Between them, ceramic and Teflon insulators were used. The hollow cathode was cooled by water. Two DC power supplies (- 500 V, 10 A and - 5 kV, 1A) were used for the hollow cathode discharge and beam acceleration, respectively. Connections of the electrical outputs of the power supplies to the electrodes are shown in **Figure 1**.

C. Langmuir probe construction, data collection and analysis

The Langmuir probe was constructed from ceramic and stainless steel tubes. The tip was a loop of tungsten wire with an exposed area of 2.5 x 10^{-3} cm^2. Langmuir probe I-V characteristics were created by plotting the measured current versus the applied bias voltage. The probe was heated at a constant current of 2.5 A to prevent oxidation and contamination of the tungsten. The Langmuir probe was operated in an emissive mode to determine the plasma potential. A LabVIEW™ program [5] was used to control the bias source (Stanford Research SR245, Keithley 2400) and to perform the data analysis. The system stepped the probe bias voltage from -15 V to 15 V in 0.02 V steps and collected and stored the probe current measured at each voltage in a master ASCII data file. A data analysis routine was used to determine the plasma electron temperature (T_e), the plasma potential (V_p), the floating potential (V_f) and the electron density (n_e). The plasma potential was determined first from the maximum of the derivative of the I-V characteristic. The probe current data was then fitted using a function of the form $I(V) = a + bV^{\gamma} + c[\exp(\varphi/Te)]$, where $\varphi = V - V_p$. The first two terms present the ion portion of the trace while the third term models the electron current. The exponent γ depends on the effective geometry of the probe (0.5 for a cylinder and 1 for a sphere), and the constants a, b and c are all parameters used to fit the data. The prefactor c is the electron saturation current, given by $c=en_eA(kT_e/2\pi m_e)^{1/2}$, where e is the electron charge, A is the probe area, k is Boltzmann's constant and m_e is the electron mass. Values of a, b and c are found for series of T_e values, with the best combination of values being the set that minimizes the error between the data and the fit. With the best c and T_e values, the electron density is finally calculated from the expression given above for c. No allowance was made for the beam current because the beam current density was much smaller than the plasma density in all experiments.

III. EXPERIMENTAL RESULTS

Spatial distributions of the electron temperature (T_e) and plasma density (n_p) in argon plasma are shown in **Figure 2**. The estimated plasma potential for argon was 2.6 V. The pressure in the chamber was 20 mTorr. The grid voltage and current were 1.3 kV and 19 mA respectively. The hollow cathode voltage and current were 360 V and 35 mA. The magnetic field strength was 100 G. The electron temperature inside the beam was 0.45 eV and was approximately constant (the variation was less than 10 %) but then fell to 0.35 eV outside the beam. By contrast, the plasma density changed by an order of magnitude (from 2.2 x 10^{10} cm^{-3} outside the beam to 2.3 x 10^{11} cm^{-3} in the middle of the beam).

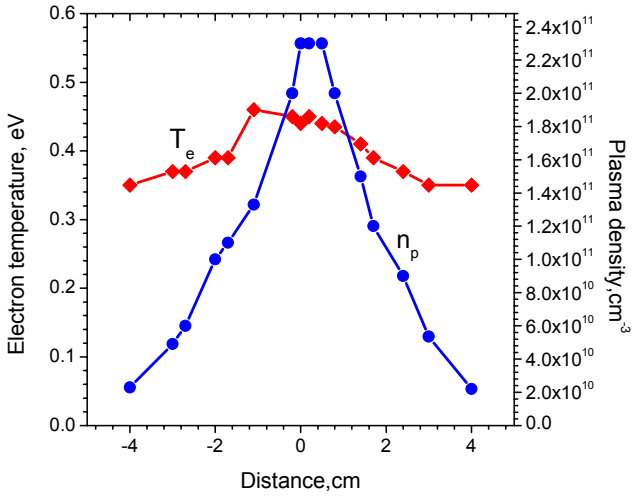

Figure 2. Spatial electron temperature (T_e) and plasma density (n_p) profiles in argon. Experimental conditions: pressure 20 mTorr, grid voltage 1.3 kV, grid current 19 mA, hollow cathode voltage 360 V, hollow cathode current 35 mA, magnetic filed strength 100 G

The effect of pressure on the electron temperature and plasma density at the beam axis is presented in **Figure 3**. The grid and the hollow cathode voltages were 1.3 kV and 320 V respectively. The hollow cathode and grid currents increased almost linearly with pressure changing from I_{HC} = 22 mA and I_g = 14 mA at 20 mTorr to I_{HC} = 51 mA and I_g = 37 mA at 50 mTorr and keeping the same currents ratio I_g/I_{HC} of 0.7. The on-axis electron temperature was 0.6 eV from 30 to 50 mTorr and then fell to 0.4 eV at 20 mTorr. The plasma density increased by almost an order of magnitude from 10^{11} cm^{-3} at 20 mTorr to 8 x 10^{11} cm^{-3} at 50 mTorr.

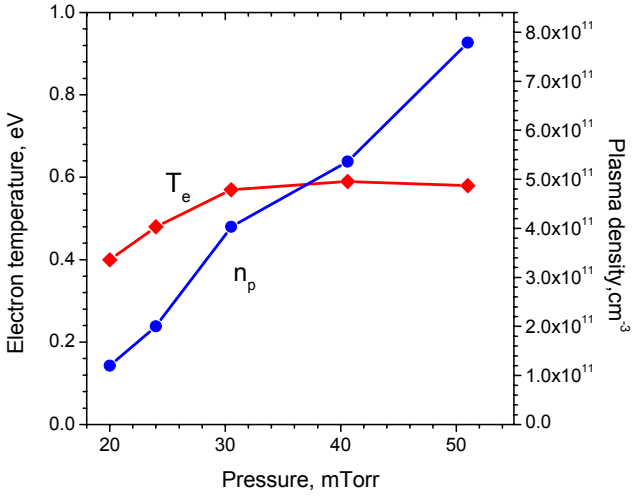

Figure 3. Effect of pressure on electron temperature (T_e) and plasma density (n_p) in Ar plasma. The measurements were performed on axis. Experimental conditions: grid voltage 1.3 kV, hollow cathode voltage 320 V, magnetic field strength 100 G.

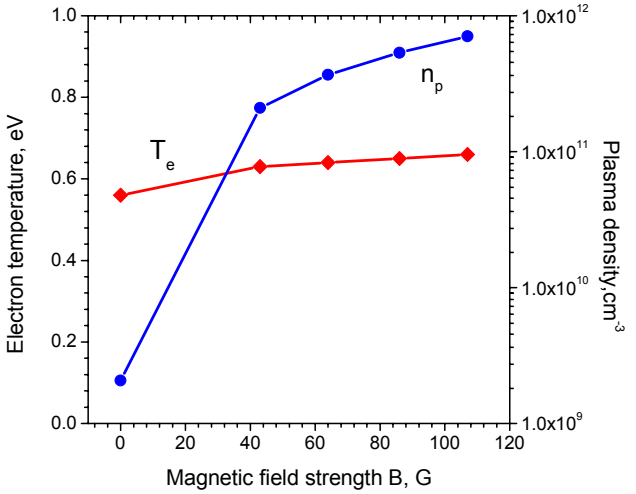

Figure 4. Effect of magnetic field on electron temperature (T_e) and plasma density (n_p) in Ar plasma. The measurements were performed on axis. Experimental conditions: pressure 50 mTorr, grid voltage 1.3 kV, hollow cathode voltage 360 V.

The magnetic field influence on electron temperature and plasma density is presented in **Figure 4**. The pressure was held constant at 50 mTorr. The grid and the hollow cathode voltages were 1.3 kV and 360 V respectively. The hollow cathode and grid currents increased with magnetic field strength almost linearly from their initial values of I_{HC} = 32 mA and I_g = 18 mA with no magnetic field to I_{HC} = 60 mA and I_g = 41 mA at magnetic field strength of 100 G and keeping almost constant current ratio I_g/I_{HC} of 0.65. As shown in Figure 4, the electron temperature was nearly constant as well, $T_e \approx 0.6$ eV. The

plasma density increased from 2 x 10^9 cm^{-3} with no magnetic field to 7 x 10^{11} cm^{-3} for a magnetic field of 110 G. The very low plasma density with no magnetic field is presumably the result of beam expansion.

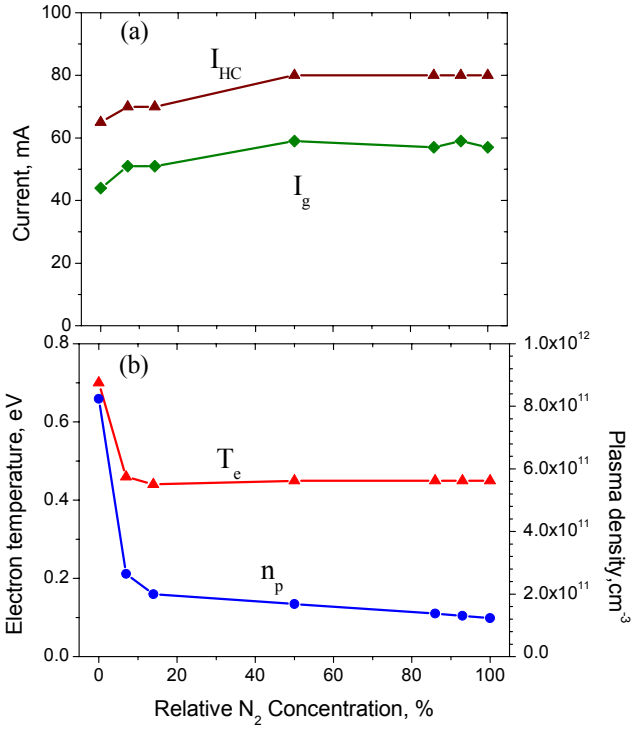

Figure 5. Effect of gas composition on hollow cathode (I_{HC}) and grid (I_g) currents (**Figure 5a**) and on electron temperature (T_e) and plasma density (n_p) (**Figure 5b**). The measurements were performed on axis. Experimental conditions: pressure 50 mTorr, grid voltage 1.3 kV, hollow cathode voltage 350 V, magnetic filed strength 100 G.

Figure 5 shows the effect of gas composition on hollow cathode and grid currents (**Figure 5a**) and on the electron temperature and plasma density (**Figure 5b**). The pressure was again 50 mTorr. The grid and the hollow cathode voltages were 1.3 kV and 350 V respectively. The hollow cathode and the grid currents changed from I_{HC} = 55 mA and I_g = 44 mA in pure argon to I_{HC} = 80mA and I_g = 60 mA in 50 % Ar and 50 % N_2 mixture and remained at those levels even in pure nitrogen. The highest electron temperature, 0.7 eV, was measured in pure argon. Only 10 % N_2 addition was sufficient to reduce the electron temperature to 0.45 eV, and T_e remained at 0.45 eV even in pure nitrogen. The plasma density was largest (8 x 10^{11} cm^{-3}) in pure argon and gradually reduced with increased nitrogen content. The lowest value of 10^{11} cm^{-3} was obtained in pure nitrogen. The plasma potential in pure nitrogen was 2 V.

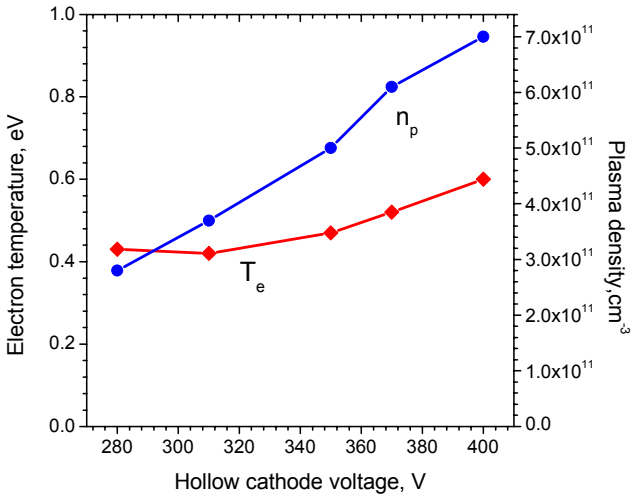

Figure 6. Effect of hollow cathode voltage variation on electron temperature (T_e) and plasma density (n_p) in Ar-N_2 mixture (93/7 vol. %). The measurements were performed on axis. Experimental conditions: pressure 50 mTorr, grid voltage 0.8 kV, hollow cathode voltage 280 V, magnetic filed strength 128 G.

The effect of the hollow cathode voltage on the electron temperature and plasma density is presented in **Figure 6**. The measurements were performed in an argon-nitrogen mixture (93 % Ar/ 7 % N_2). In this experiment the pressure was 50 mTorr, the grid and hollow cathode voltages were 0.8 kV and 280 V respectively. The magnetic field strength was 130 G. The hollow cathode and grid currents increased almost linearly with hollow cathode voltage from their initial values of I_{HC} = 40 mA and I_g = 36 mA at hollow cathode voltage of 280 V to I_{HC} = 130 mA and I_g = 113 mA at hollow cathode voltage of 400 V. For this currents range the plasma density increased more than 2 times from 3 x 10^{11} cm^{-3} to 7 x 10^{11} cm^{-3}. The electron temperature also changed from 0.43 eV to 0.6 eV.

IV. DISCUSSION

The experimental results show that over the investigated parameters range in pure noble, pure molecular gases, and their mixtures the electron temperature was less than 1 eV. The plasma density in the beam was in order of 10^{11} cm^{-3} when beam was collimated by magnetic field. In argon, the electron temperature was almost constant across the beam width, which is consistent with the absence of a significant potential drop within the ionization region. However, a voltage drop is expected on the far sides of the plasma and there the electron temperature fell. A magnetic field was applied along the beam direction (z-axis) to confine the beam to a thin sheet. But the magnetic field retards the outward flow of plasma electrons even more, especially in argon, which has a strong Ramsauer minimum [6]. In this

case electron pressure must overcome the magnetic force as well as ion drag, and therefore the electron temperature increased slightly when the magnetic field was introduced. The plasma density increased even more because the field retards diffusion.

The gas pressure and the hollow cathode voltage strongly influenced the plasma density but affected the electron temperature only weakly. In particular, raising the gas pressure or hollow cathode voltage caused the beam current density to rise due to an increase in hollow cathode plasma density. The plasma density and electron temperature in the beam produced plasma increased in response. The temperature variation was relatively modest, however.

The only parameter that strongly influenced the electron temperature was the gas composition. Even a slight addition of molecular gas (N_2) was sufficient to reduce the electron temperature significantly. The effect can be understood by recognizing that noble and molecular gases differ in two key ways. First, the major electron loss mechanism in noble gases is ambipolar diffusion to the walls, whereas in molecular gases electron attachment and recombination processes are usually more important. The latter is especially important for the plasmas studied here. In addition, unlike molecular gases, noble gases lack low-lying excited states. For example, the lowest excited state in argon is a metastable level at 11.55 eV (as compared with the ionization energy of 15.8 eV), and therefore collisional cooling effectively ceases at energies below 11.55 eV. As a result, the electrostatic plasma field extracts energy faster than collisions.

Low electron temperature is a unique characteristic of electron beam generated plasmas and it affects the material processing in several ways. First, in the absence of any external bias, the ion energy incident on a material is much lower (~ 2 eV) compared to the other sources, and therefore surface roughening or other damage to sensitive materials is reduced. Second, when a bias is applied to raise the ion energy, the spread in energy is less, and consequently specific surface reactions can be controlled better. Lastly, in many halogen-based gases, large negative ion densities [7] can be produced, when the electron temperature is low. As a result, dense ion-ion plasmas can be generated continuously, a feature not available with discharges.

V. SUMMARY

We have described analysis of plasma parameters of continuous electron beam-generated plasma source based on Langmuir probe diagnostics. The effects of gas pressure, magnetic field, hollow cathode voltage and gas composition on the plasma generation were investigated. Gas composition was the only parameter that greatly influenced the electron temperature due to different electron loss mechanisms. The highest electron temperature of 0.7 eV was measured in pure argon. Plasma densities in the beam were in the 10^{11} cm^{-3} range, which is sufficient for material processing applications. Further studies are on the way to characterize the types and energy of the ions generated in this plasma source.

VI. REFERENCES

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